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## PATENT SPECIFICATION

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### (54) METHOD OF PRODUCING VEGETABLE FATS AND OILS

#### SPECIFICATION NO 1356749

By a direction given under Section 17 (1) of the Patents Act 1949 this application proceeded in the name of STUDIENGESELLSCHAFT KÖHLE M.B.H., a Company incorporated under the laws of Germany, of Kaiser-Wilhelm-Platz 1, D-4330 Mulheim, Germany.

THE PATENT OFFICE

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Known methods of producing vegetable fats and oils which have been used industrially include production by mechanical expressing, production by extraction, and production by boiling steaming, or melting out.

15 Of these the first two are most often used. While by pressing it is impossible to obtain the entire fat content of the vegetable, this can be dissolved out of the material by extraction 20 with a residue of less than 1%.

All these methods require pre-treatment, which in some cases is very extensive and includes comminution and shaping the vegetable material. In extraction processes the 25 residues of solvent in the fat and in the extraction residue, which in some cases is used for fodder purposes, may constitute a considerable problem. Residues of solvent are naturally very difficult to remove completely, and consequently 30 the problem of their removal is receiving considerable technical and industrial attention. For these reasons in particular there is a complete ban on solvent extraction processes for fats in some countries, in which case only pressing methods, with consequently poorer yields, 35 may be allowed.

The main object of the present invention is 40 to provide an improved method of producing vegetable fats and oils, particularly seed fats. It is based on the observation that gases which 45 are supercritical in respect of both pressure and temperature and which are unobjectionable chemically and in respect of health and taste are a solvent for vegetable fats and oils.

45 According to the present invention, a

Carbon dioxide is preferred as the extraction medium, but SF<sub>6</sub>, CHF<sub>3</sub>, CF<sub>3</sub>Cl, Cl<sub>2</sub>=CH<sub>2</sub>, CHF<sub>2</sub>Cl, C<sub>2</sub>F<sub>6</sub> and N<sub>2</sub>O, which is isosteric with CO<sub>2</sub>, and also ethane or ethylene, all of which are likewise gases which are unobjectionable chemically and in the respect of taste and health, may also be satisfactorily used, either alone or mixed with one another and/or with carbon dioxide after they have been subjected, if necessary or desirable to pre-purification, such as for example by distillation. The preferred working conditions indicated in the four cases explained below may be applied appropriately to the gases mentioned above, in respect of their critical data, which are known.

It should be observed that extraction with CO<sub>2</sub> is also possible in the liquid, that is to say sub-critical, state, and this moreover also applies to the other gases mentioned above. When the supercritical state is reached, however, solvent power changes abruptly and in that condition is considerably greater. With the method of the invention it is possible for the fats or oils to be completely extracted in their natural composition from the vegetable material without chemical modification even of the residue.

In order that the invention may be more fully understood an embodiment in accordance therewith will now be described by way of example with reference to the accompanying drawing which shows, diagrammatically an apparatus employed in carrying out the process

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of the invention. In this example carbon dioxide is used as the extraction solvent gas, but it is to be understood that other suitable solvent gases, for example those named above, or mixtures thereof, could be used in place of the carbon dioxide, appropriate changes being made to take account of different critical pressures and temperatures of such a solvent gas.

Referring to the drawing, a vessel A is charged with crushed or coarsely ground material. Neither special shaping of the material nor the removal of husks or shells is necessary, provided that the husks or shells contain no constituents which may be extracted and which are not desirably present in the fat or oil. After removal of atmospheric oxygen by flushing out the entire installation with  $\text{CO}_2$ , supercritical  $\text{CO}_2$  from a gas storage G is introduced into the vessel A and is there charged with the vegetable fat in the form of a "supercritical solution".

The vessel is closed with fine-pored metal filter discs (filter threshold 3—5  $\mu$ ), so that mechanical entrainment of parts of the plants is not possible. The gas current is then transferred through the heat exchanger W 3 to a vessel B, where the mixture is separated. This is preferably effected by lowering the pressure to values less than  $P_{\text{crit}}$ , gas. The temperature may likewise be lowered to below  $t_{\text{crit}}$ , gas, left at the same level as that in the extraction vessel A, or may be raised.

In principle the raising of the temperature will also lead to the separation of the system, similarly to the lowering of the pressure below  $P_{\text{crit}}$ . The two effects may be combined, but in this case one should be guided by the thermal stability of the extract. This results in a number of variants of the method, which will be indicated herein.

If the temperature of the charged current of gas on entering the vessel B is lowered below  $t_{\text{crit}}$  during the separation of the mixture, liquefaction of the  $\text{CO}_2$  will occur. It has been found that in this case, in addition to the separation of the extract, i.e. of the fat or oil, the

water originating from the vegetable material will also be separated from the mixture. Since the water fraction is not generally desired in the fat or oil, it is preferable to carry out the method so that the mixture separation temperature in B does not fall below  $t_{\text{crit}}$  but remains higher. This will result in the removal of the fat or oil components from the mixture, but only very small amounts of water will also be removed from the mixture, so that a fat or oil practically free from water is obtained. The fat or oil can be removed from the vessel B on completion of the extraction by raising the temperature to values above the clear melting point, lowering the pressure in vessel B to a few atmospheres gauge, and removing the fat or oil in liquid form. The dissolved  $\text{CO}_2$  is thereby also separated from the mixture, and at the same time acts as an inert gas.

The current of gas free from the extract leaves the vessel B, and is conveyed and compressed by means of a liquid gas pump or compressor K, and passes back to the extraction vessel A as a pure solvent, and so on. Depending on the choice of the conveyer, the heat exchangers W1 and W2 have different functions. In the case of conveyance by the liquid gas pump, liquefaction of the  $\text{CO}_2$  in the heat exchanger W 2 is necessary, while the medium must also be heated to supercritical conditions in the heat exchanger W 1. When a compressor is used, the medium must if necessary be brought to supercritical temperatures in the heat exchanger W 2, while the heat of compression must be removed in W 1. In practice the heat exchangers are connected together, so that economical operation is possible in respect of energy requirements. As mentioned, a number of variants of the method are possible by applying different combinations of the parameters, pressure and temperature, in the separation of the mixture, where  $P_1$  and  $t_1$  apply to pressures and temperatures in the extraction vessel A, and  $P_2$  and  $t_2$  apply to the mixture vessel B:

First case: Conditions:  $P_2 < P_{\text{crit}} < P_1$ ,

$t_2 < t_{\text{crit}} < t_1$

Preferred absolute values

$P_1 = 100-400$  atmospheres  
gauge

$P_2 = 5$  atmospheres below  $P_{\text{crit}}$

$t_1 = 5^\circ$  above  $t_{\text{crit}}$  up to  $100^\circ\text{C}$ .

$t_2 = 5^\circ\text{C.}$  below  $t_{\text{crit}}$

The  $\text{CO}_2$  is liquefied in B, and consequently a certain proportion of water is also separated.

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Second Case: Conditions:  $P_2 = P_1 > P_{crit}$

$t_2 > t_1 > t_{crit}$

preferred absolute values

$P_1 = 100-400$  atmospheres  
gauge

$t_1 = \text{see Case 1}$

$t_2 = 40-80^\circ\text{C}$ . higher than  $t_1$

Because  $t_2$  is higher than  $t_1$ , this case is possible only when permitted by the thermal stability of the extract.

Third Case: Conditions:  $P_2 < P_{crit} < P_1$

$t_2 = t_1 > t_{crit}$

preferred absolute values:

$P_1, P_2, t_1, t_2 = \text{as in Case 1}$

Fourth Case: Conditions:  $P_2 < P_{crit} < P_1$

$t_2 > t_1 > t_{crit}$

preferred absolute values:

$t_2 = \text{see Case 2}$

$P_1, P_2, t_1, t_2 = \text{as in Case 1}$

Because  $t_2$  is higher than  $t_1$ , the remarks made in connection with Case 2 are also applicable here.

5 The method is carried out for between 1 and 8 hours, depending on parameters.

With the aid of the method of the invention more than 99% of the extractable fat and oil contents can be removed from the 10 seeds; in addition, with the aid of this method

it is possible to obtain fats and oil which are practically free from water.

The invention will now be further described with reference to the following Examples.

20 Gas: dry  $\text{CO}_2$   
Time: 3 hours

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$P_1 = 350$  atmospheres gauge  
 $t_2 = 50^\circ\text{C}$   
 $P_2 = 65$  atmospheres gauge  
 $t_1 = 50^\circ\text{C}$

Results:

25 354 g of a solid whitish yellow coconut fat were obtained. The residue was a dry, light brown, powdery mass. Analytical data: see summarising table.

Gas: dry  $\text{CO}_2$   
Time: 4 hours

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Example 1.

600 g of coarsely crushed copra were processed in the installation shown in the drawing under the conditions of Case 3, as follows:

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$P_1 = 350$  atmospheres gauge  
 $t_2 = 50^\circ\text{C}$   
 $P_2 = 65$  atmospheres gauge  
 $t_1 = 55^\circ\text{C}$

Example 2.

600 g of sunflower seeds were coarsely crushed and processed in the installation in accordance with the conditions of Case 4, as follows:

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Results:  
40 148 g of a light yellow, clear oil having a pleasant odour were obtained. The residue left was a dry, crumbly mass. Analytical data: see summarising table.

Gas: dry  $\text{CO}_2$   
Time: 5 hours

500 g of coarsely crushed soya beans were processed in the installation in accordance with the conditions of Case 4, as follows:

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$P_1 = 280$  atmosphere gauge  
 $t_2 = 45^\circ\text{C}$   
 $P_2 = 56$  atmospheres gauge  
 $t_1 = 60^\circ\text{C}$

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## Results:

112 g of a light yellow oil with a mild, pleasant aroma was obtained. The residue left was a dry, light yellow, powdery mass.

5 Analytical data: see summarising table.

10 Gas: dry CO<sub>2</sub>  
Time: 3 hours

## Example 4.

450 g of coarsely crushed peanut kernels were processed in the installation in accordance with the conditions of case 1, as follows:

$$\begin{aligned}P_1 &= 300 \text{ atmospheres gauge} \\t_1 &= 50^\circ\text{C} \\P_2 &= 56 \text{ atmospheres gauge} \\t_2 &= 20^\circ\text{C}\end{aligned}$$

## 15 Results:

235 g of a light yellow oil with a peanut aroma, which was made slightly turbid due to a slight water content, were obtained. The

residue left was a dry light yellow, granular mass.

Analytical data: see summarising table

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Analytical data:

Samples	$n_{D}^{20}$	$n_{D}^{20}$	Saponification number VZ	Acid number SZ	Iodine number IZ	Fat content % referred to the dry state	Water Content %	Clear melting point °C.
Copra, unprocessed						62.5	3.6	
Coconut fat, extracted	1.4490		259	2.5	8.4		<0.1	25.2
Copra processed						0.5	8.8	
Comparative values from literature for coconut fat	1.418— 1.450		256—262		7—I0			22—55
Sunflower seeds unprocessed						25	2.5	
Sunflower seed oil, extracted	1.4675		192	3.5	130		<0.1	
Sunflower seeds, Processed						0.4	2.9	
Comparative values for sunflower seed oil from literature	1.4660— 1.4680		188—194		125—140			

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Analytical data (Continued):

Soya beans, unprocessed					19.2	3.9
Soya bean oil extracted	1.4674	189	2.6	125	<0.1	
Soya beans processed					0.6	4.8
Comparative values for soya oil from literature	1.4650— 1.4690	189—195		117—141		
Peanut kernels, unprocessed					54.4	2.7
Peanut oil, extracted	1.4614	1.4707	190	1.9	93	<0.1
Peanut kernels processed					2.2	5.5
Comparative values for peanut oil from literature	1.4700— 1.4710	184—195		84—95		

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**WHAT WE CLAIM IS:—**

1. A method of producing vegetable fats and oils by extraction with a solvent, wherein the fat or oil is removed from the vegetable material by extraction with supercritical gases comprising SF<sub>6</sub>, CHF<sub>3</sub>, CHF<sub>2</sub>Cl, CF<sub>3</sub>Cl, CF=CH<sub>2</sub>, C<sub>2</sub>F<sub>5</sub>, N<sub>2</sub>O, ethane, ethylene or CO<sub>2</sub>, or a mixture of any two or more thereof, the fat or oil being thereafter separated from the resulting solution by modifying the pressure and/or temperature.

2. A method according to Claim 1 in which the separation is effected by lowering the pressure to below the critical pressure and maintaining the temperature above the critical temperature.

3. A method according to Claim 1, in which

the separation is effected by lowering the pressure to below the critical pressure and lowering the temperature to below the critical temperature.

4. A method according to Claim 1 and substantially as hereinbefore described with reference to the drawing.

5. A method according to Claim 1 and substantially as described in any of the Examples.

6. A vegetable fat or oil whenever produced by a method according to any of the preceding Claims.

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COMPLETE SPECIFICATION

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This drawing is a reproduction of  
the Original on a reduced scale

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